

SLOVENSKI STANDARD
oSIST prEN ISO 3104:2018
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Naftni proizvodi - Prozorne in neprozorne tekočine - Določevanje kinematične viskoznosti in izračun dinamične viskoznosti (ISO/DIS 3104:2017)

Petroleum products - Transparent and opaque liquids - Determination of kinematic viscosity and calculation of dynamic viscosity (ISO/DIS 3104:2017)

Mineralölerzeugnisse - Durchsichtige und undurchsichtige Flüssigkeiten - Bestimmung der kinematischen Viskosität und Berechnung der dynamischen Viskosität (ISO/DIS 3104:2017)

Produits pétroliers - Liquides opaques et transparents - Détermination de la viscosité cinématique et calcul de la viscosité dynamique (ISO/DIS 3104:2017)

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ICS:

75.080	Naftni proizvodi na splošno	Petroleum products in general
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Petroleum products — Transparent and opaque liquids — Determination of kinematic viscosity and calculation of dynamic viscosity

*Produits pétroliers — Liquides opaques et transparents — Détermination de la viscosité cinématique et
calcul de la viscosité dynamique*

ICS: 75.080

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Foreword

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

The committee responsible for this document is ISO/TC 28, *Petroleum and related products, fuels and lubricants from natural or synthetic sources*.

This third edition cancels and replaces the second edition (ISO 3104:1994), of which it constitutes a technical revision. Major change is that the precision data are updated to all actual fuels on the market; biodiesel (FAME) blends and paraffinic diesel have been included in the scope. Another important change is that the procedure description and allowance of automated techniques have been included.

Introduction

Many petroleum products, and some non-petroleum materials, are used as lubricants, and the correct operation of equipment depends upon the appropriate viscosity of the liquid being used. In addition, the viscosity of many petroleum fuels is important for the estimation of optimum storage, handling and operational conditions. Thus the accurate measurement of viscosity is essential to many product specifications.

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Petroleum products — Transparent and opaque liquids — Determination of kinematic viscosity and calculation of dynamic viscosity

1 Scope

This International Standard specifies a Procedure A using manual glass viscometers and a Procedure B using glass capillary viscometers in an automated assembly, for the determination of the kinematic viscosity, ν , of liquid petroleum products, both transparent and opaque, by measuring the time for a volume of liquid to flow under gravity through a calibrated glass capillary viscometer. The dynamic viscosity, η , is obtained by multiplying the measured kinematic viscosity by the density, ρ , of the liquid. The range of kinematic viscosities covered in this test method is from (0,2 to 300 000) mm²s over the temperature range (– 240 to + 150)°C.

NOTE The result obtained from this International Standard is dependent upon the behaviour of the sample and is intended for application to liquids for which primarily the shear stress and shear rates are proportional (Newtonian flow behaviour). If, however, the viscosity varies significantly with the rate of shear, different results may be obtained from viscometers of different capillary diameters. The procedure and precision values for residual fuel oils, which under some conditions exhibit non-Newtonian behaviour, have been included.

WARNING — The use of this Standard can involve hazardous materials, operations and equipment. This Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of users of this standard to take appropriate measures to ensure the safety and health of personnel prior to the application of the standard, and fulfil statutory and regulatory requirements for this purpose.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3105:1994, *Glass capillary kinematic viscometers — Specifications and operating instructions*

ISO/TR 3666, *Viscosity of water*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

ASTM E1137, *Standard Specification for Industrial Platinum Resistance Thermometers*

ASTM E2877, *Standard Guide for Digital Contact Thermometers*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1 kinematic viscosity

ν

resistance to flow of a fluid under gravity

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Note 1 to entry: For gravity flow under a given hydrostatic head, the pressure head of a liquid is proportional to its density, ρ . For any particular viscometer, the time of flow of a fixed volume of fluid is directly proportional to its kinematic viscosity, ν , where $\nu = \eta/\rho$, and where η is the dynamic viscosity coefficient.

3.3

dynamic viscosity η

ratio between the applied shear stress and rate of shear of a liquid, sometimes called the coefficient of dynamic viscosity, or simply viscosity; it is a measure of the resistance to flow or deformation of a liquid

Note 1 to entry: The term dynamic viscosity is also used in a different context to denote a frequency-dependent quantity in which shear stress and shear rate have a sinusoidal time dependence.

3.3

density ρ

mass per unit volume of a substance at a given temperature

4 Principle

The time is measured for a fixed volume of liquid to flow under gravity through the glass capillary of a calibrated viscometer under a reproducible driving head and at a known and closely controlled temperature. The kinematic viscosity is the product of the measured flow time and the calibration constant of the viscometer.

5 Reagents and materials

5.1 Cleaning solution, strongly-oxidizing cleaning solution or alkaline cleaning solutions can be used.

5.2 Sample solvent, completely miscible with the sample. Filter before use.

NOTE For most samples a volatile petroleum spirit or naphtha is suitable. For residual fuels, a prewash with an aromatic solvent such as toluene or xylene may be necessary to remove asphaltenic material.

5.3 Drying solvent, volatile and miscible with both the sample solvent (5.2) and water (5.4). Filter before use.

NOTE Acetone is suitable.

5.4 Water, deionized or distilled, conforming to Grade 3 of ISO 3696. Filter before use.

5.5 Certified viscosity reference standards, (CRM) – data provided by an accredited calibration laboratory - traceable to the international agreed value of distilled water (1,003 4 mm²/s at 20 °C) as specified in ISO/TR 3666 and calibrated in accordance with a standard practice for the basic calibration of master viscometers and viscosity oils, such as in ASTM D2162^[1].

6 Apparatus design and requirements

6.1 Drying tubes, consisting of a desiccant drying system, consisting of either externally mounted drying tubes or an integrated desiccant drying system designed to remove ambient moisture from the capillary tube. Ensure that they are packed loosely and that the silica gel is not saturated with water.

6.2 Sample filter, micron screen or fretted (sintered) glass filter, no more than 75 μm .

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6.3 Reagent filter, micron screen or fretted (sintered) glass filter, no more than 11 μm .

6.4 Ultrasonic bath, unheated – with an operating frequency between 25 kHz to 60 kHz and a typical power output of ≤ 100 W, of suitable dimensions to hold container(s) placed inside of bath, for use in effectively dissipating and removing air or gas bubbles that can be entrained in viscous sample types prior to analysis. It is permissible to use ultrasonic baths with operating frequencies and power outputs outside this range; however it is the responsibility of the laboratory to conduct a data comparison study to confirm that results determined with and without the use of such ultrasonic baths does not materially impact results.

6.5 Manual apparatus

6.5.1 Glass capillary viscometer, calibrated in accordance with ISO 3105.

The viscometer shall have a certificate of calibration provided by an accredited laboratory.

The calibration constant, C , is dependent upon the gravitational acceleration at the place of calibration and this shall therefore be supplied by the viscometer calibration laboratory, together with the instrument constant. The variation in the value of g across the earth's surface is about 0,5 % due to latitude plus approx. 0,003 % per 100 m altitude. Apply a gravity correction to the viscometer calibration constant if the acceleration of gravity of the testing laboratory differs by more than 0,1% of the calibration laboratory.

$$C_2 = (g_2/g_1)C \quad (1)$$

where the subscripts 1 and 2 indicate, respectively, the calibration laboratory and the testing laboratory

NOTE Calculation of acceleration of gravity values can be found at www.NPL.co.uk.

IMPORTANT — Viscometers used for silicone fluids, fluorocarbons and other liquids, which are difficult to remove by the use of a cleaning agent, shall be reserved for the exclusive use of those fluids, except during their calibration. Subject such viscometers to calibration checks at frequent intervals. The solvent washings from these viscometers shall not be used for the cleaning of other viscometers. If the viscometer is cleaned using the material in 5.1 then the user shall verify the calibration before further use.

6.5.2 Viscometer holder or mounting device within the temperature controlled bath, enabling the glass viscometer to be suspended so that the upper meniscus is directly above the lower meniscus vertically within 1 ° in all directions.

Those viscometers whose upper meniscus is offset from directly above the lower meniscus shall be suspended vertically within 0,3 ° in all directions (see ISO 3105).

The proper alignment of vertical parts may be confirmed by using a plumb line, but for rectangular baths with opaque ends this may not be possible.

6.5.3 Temperature-controlled bath, containing a transparent liquid of sufficient depth such that at no time during the measurement is any portion of the sample in the viscometer less than 20 mm below the surface of the bath liquid or less than 20 mm above the bottom of the bath.

Temperature control of the bath liquid shall be such that, for each series of flow-time measurements, within the range of 15 °C to 100 °C the temperature of the bath medium does not vary by more than \pm

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0,02 °C from the selected temperature over the length of the viscometer, or between the position of each viscometer, or at the location of the thermometer. For temperatures outside this range, the deviation from the desired temperature shall not exceed $\pm 0,05$ °C.

6.5.3.1 Adjust and maintain the viscometer bath at the required test temperature within the limits given in 6.5.3, taking account of the conditions given in Annex B and of the corrections supplied on the certificates of calibration for the thermometers. Maintain the bath temperature at the test temperature using the readings of the temperature measuring device with the corrections supplied by the certificate of calibration.

Thermometers shall be held in an upright position under the same conditions of immersion as when calibrated. In order to obtain the most reliable temperature measurement, it is recommended that two thermometers with valid calibration certificates be used. They should be viewed with a lens assembly giving approximately 5x magnification and be arranged to eliminate parallax errors.

6.5.4 Temperature-measuring device, for the range 0 °C to 100 °C, being either

- a) a calibrated liquid-in-glass thermometer, (Annex B) with a calibration and measurement capability (CMC) of $\pm 0,02$ °C after correction or better, or
- b) a digital contact thermometer as described in 6.5.6.1 with equal or better CMC.

The calibration data should be traceable to a calibration or metrology standards body and meet the uncertainty of measurement required. The calibration certificate shall include data covering the series of temperature test points which are appropriate for its intended use. When two thermometers are used in the same bath in this range, they shall agree within 0,04 °C. See Annex B for the list of complying thermometers.

If calibrated liquid-in-glass thermometers are used, the use of two thermometers is recommended.

Outside the range 0 °C to 100 °C, a calibrated liquid in-glass thermometer (Annex B) or a digital contact thermometer as described in 6.5.4.1 with a calibration and measurement capability (CMC) of $\pm 0,05$ °C or better shall be used, and when two thermometers are used in the same bath they shall agree within $\pm 0,1$ °C.

When using liquid-in-glass thermometers, use a magnifying device to read the thermometer to the nearest 1/5 division (for example, 0,01 °C or 0,02 °F) to ensure that the required test temperature and temperature control capabilities are met. It is recommended that thermometer readings (and any corrections supplied on the certificates of calibrations for the thermometers) be recorded on a periodic basis to demonstrate compliance with the test method requirements.

6.5.4.1 Digital contact thermometer (DCT) meeting the requirements in Table 1.

NOTE The resulting uncertainty of calibration may be dependent upon the immersion depth

6.5.4.2 The DCT probe is to be immersed no less than the immersion depth stated on the calibration certificate.

NOTE With respect to DCT probe immersion depth, a procedure is available in ASTM E644, Section 7 [2], for determining the minimum depth. With respect to an ice bath, ASTM E563^[3] provides guidance on the preparation of an ice bath however variance from the specific steps is permitted provided preparation is consistent as it is being used to track change in calibration.